

Standard Operating Procedure for Perkin Elmer CHN Analyzer (Model 2400)

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This is an appendix to the EPA Lake Michigan Mass Balance QAPP for GLERL Sediment trap and Sediment samples

Items in *italics* are instrument control panel functions

For more detailed information, see the instrument manual provided by Perkin Elmer.

1.0 Initialization

1.1 Check that furnace is on (*parameter 12*)

1.2 Check oven temperatures

1.2.1 Combustion oven 925° C (*parameter 7*)

1.2.2 Reduction oven 640° C (*parameter 8*)

1.3 Check pressure gauges

O₂ 15 psi

He 20 psi

Air 60 psi

1.4 Purge He and O₂ gases when:

1.4.1 Gases have been changed

1.4.2 Reduction or combustion tubes have been changed

1.4.3 Instrument has been down a long time

He 120 seconds

O₂ 20 seconds

1.5 Run a combustion zone leak test

Enter *diagnostics*

Gas

Leak test

Combustion zone leak test -2

1.5.1 Pressure will rise to above 760 psi and stabilize. To pass, it should stay above 760 psi. Enter *diagnostics* to get out of diagnostic mode, when the test has been completed.

- 1.6 Release H-valve to reduce pressure caused by the combustion zone leak test.
- Enter *Diagnostic*
Gas
Valve
7-on (wait approx. one minute)
Enter *diagnostic* to close valve and get out of diagnostic mode.
- 1.7 List parameters
- Hit *Monitor* key, press *monitor* key again to go to standby.
- 1.8 Calibrate autobalance - repeat three times, see autobalance manual.
- 1.9 Sediment trap Sample Preparation (after freeze drying)
- 1.9.1 Separate each interval of the core or sediment trap sample/composite into an unacidified (total carbon and nitrogen) segment, and acidified (organic carbon and nitrogen) segment.
- 1.9.2 Unacidified segment - grind and dry @ 80-90°C.
- 1.9.3 Acidified segment - add 1N HCl, shake overnight, dry and regrind.
- 1.9.4 Weigh each sediment interval within a tin capsule, which must be tared before weighing the sediment.
- 1.9.5 It's critical to follow sterile handling procedures with the tweezers (all handling instruments), and the aluminum foil workspace. Wipe instruments and workspace with tissue paper between samples.
- 1.9.6 Fold tin capsule so as to easily pass through the CHN entry chamber hole without getting caught by its edges.
- 1.10 Run five instrument blanks
- 1.10.1 Hit *single run* - press blank then 5.
- 1.10.2 Blanks should reproduce within the following range:
- | | |
|----------|------|
| Carbon | ±30 |
| Hydrogen | ±100 |
| Nitrogen | ±16 |

- 1.11 Standardize instrument - sets groundwork for the K-factors.
- 1.11.1 Weigh four acetanilide samples between 2-3 mg
- 1.11.2 Run three as K-factors; should get three K-factors within tolerance.
Hit *single run*, K-factor then S1 and weight.
- | | |
|----------|----------|
| Carbon | 16 ± 3.5 |
| Hydrogen | 50 ± 20 |
| Nitrogen | 6 ± 3 |
- 1.11.3 Run acetanilide as a sample. Certification that instrument is within tolerances. ($\pm 2\%$ acetanilide). Run over if not within tolerance.
- | | | |
|----------|-------|---|
| Carbon | 71.09 | % |
| Hydrogen | 6.7 | % |
| Nitrogen | 10.36 | % |
- 1.11.4 Run two tin blanks.
- 1.12 Single run vs Autorun
- 1.12.1 *Single run mode*: Run samples one at a time. Hit *single run*, then sample; enter ID and corresponding weight.
- 1.12.2 *Autorun mode*: Run up to 60 consecutive samples in a carousel. Load samples within carousel, hit *autorun* key. Press 4 RP (reset-print), then 1 Reset; this will reset internal counter to 1 which coincides with the first slot in the carousel. Enter the ID number and weight of the first sample, press enter then start. This starts the autocarousel, then continue to enter ID and weight for the remaining samples.
- 1.13 Criteria for acceptance of data
- 1.13.1 Run standard as samples every 10 samples, result should be within 2 sd of average value.
- 1.13.2 Number of counts for Nitrogen (NR-ZR) >100 or sample was too small and the performance of the instrument will not be acceptable.
- 1.14 Shut off the He tank at the end of the day.

